REPORT

Required under the terms of NASA Research Grant NsG 376

"Theoretical and Experimental Studies
of the Underlying Processes and Techniques
of Low Pressure Measurement"

June 1, 1966 to November 30, 1966

December 1, 1966 to May 31, 1967

Personnel:

R. N. Peacock
F. M. Propst
J. W. Culton
W. C. Schuemann
D. E. Coad
G. G. Tibbetts

I. Schuemann Gauge Development

The program of Schuemann gauge refinement started during the last period has been continued. It is the aim of this work to make the gauge a more practical instrument for measuring very low pressures to perhaps 10^{-13} Torr.

A total of twelve gauges have been built and tested or, in some cases, are still under test (See Fig. 1). The gauges have been designed to overcome certain objections to the former models, namely continued outgassing of the gauge materials and difficulty of heating by electron bombardment. Recent models of this gauge have:

(1) A large spherical bulb to help keep the walls cool during outgassing, and also during normal operation at low pressure.

(2) Separate filaments, one of pure tungsten, one of thoriated tungsten. Use of the latter, low temperature cathode is necessary if extremely low pressures are to be observed.

(3) A collector shield closed at the top to eliminate the influence of stray wall potentials, and also to reduce photo emission from the upper surface resulting from scattered x-rays.

(4) A conducting tin oxide film on the inner surfaces of the bulb. This may not be necessary for future gauges, but has been very helpful in understanding the importance of wall potentials and their relation to operating conditions.

Four of these gauges from a recent batch will be used on the research described in Section III of this report.

II. Low Pressure Absolute Manometer

A feasibility study has been made for a new type of absolute pressure gauge capable of detecting pressures as low as 10^{-12} Torr. All components of the gauge are inert and compatible with UHV techniques. The gauge uses technology which already exists for molecular beam measurements in addition to some new ideas. The two main applications for this gauge would be the absolute calibration of ionization or other gauges below 10^{-4} Torr, or use as the primary pressure gauge for ionization cross section measurements. In addition, it is possible that the inert nature of the gauge would make accurate sticking probability experiments possible.

At the present time there is no satisfactory absolute gauge for use at pressures below 10^{-5} Torr. The McLeod gauge is the most commonly used instrument in the pressure range of 10^{-3} Torr to 10^{-5} Torr. Available publications state that an accuracy of 5% at 10^{-5} Torr is possible with the McLeod. The variation of published values for ionization cross sections, where the McLeod was used for the pressure calibration, suggests that the actual accuracy is 20% to 30% at 10^{-5} Torr.

The basic device, which is illustrated in Fig. 2, consists of a metallic vane mounted on the end of an arm suspended at the center of a torsion fiber. The unknown pressure is on one side and a high vacuum on the other side of the vane. Molecules rebounding from the vane give rise to a torque about the axis of the torsion fiber. A conducting grid is placed in front of the vane and a torque, opposing that caused by the molecules, can be produced by applying a voltage between the grid and the vane. A mirror is placed on the arm at the torsion fiber and optical techniques are used to determine a null position. The gas pressure on the back of the vane is made negligible by placing the vane in a wall between two vacuum systems. The first system contains the gas to be measured and the grid to produce the electrostatic force. The second system contains the suspension fiber mounting and a pump with pumping speed very much larger than the leakage conductance from system 1 to 2 through the gap around the vane.

The vane is made co-planar with the wall at the null position and the grid is parallel to the wall giving a uniform electric field. The electrostatic force then is proportional to the square of the voltage. The minimum usable voltage is probably about 0.1 volts, since contact potential differences could be of this magnitude. The maximum usable voltage is about 1000 volts in order to avoid field emission or breakdowns. This gives a pressure range of 10^{-8}, or from 10^{-4} to 10^{-12} Torr. There is no reason why the absolute accuracy could not be better than 1% over this entire range because all of the variables are known, can be measured, or calculated exactly.

These are:

(1) Voltage between vane and grid.

(2) Electrostatic field from voltage and geometry.

(3) System temperature.

(4) Vane null position with no pressure differential.

(5) Approximate pressure on the back of the vane.

The above assumes steady state operation. In nonequilibrium situations, the accuracy of the gauge would suffer because of a net gas flow onto or off of the surface of the vane.

determine a null position. The gas pressure on the back of the vane is made negligible by placing the vane in a wall between two vacuum systems. The first system contains the gas to be measured and the grid to produce the electrostatic force. The second system contains the suspension fiber mounting and a pump with pumping speed very much larger than the leakage conductance from system 1 to 2 through the gap around the vane.

The vane is made co-planar with the wall at the null position and the grid is parallel to the wall giving a uniform electric field. The electrostatic force then is proportional to the square of the voltage. The minimum usable voltage is probably about 0.1 volts, since contact potential differences could be of this magnitude. The maximum usable voltage is about 1000 volts in order to avoid field emission or breakdowns. This gives a pressure range of 10^{-8}, or from 10^{-4} to 10^{-12} Torr. There is no reason why the absolute accuracy could not be better than 1% over this entire range because all of the variables are known, can be measured, or calculated exactly.

These are:

(1) Voltage between vane and grid.

(2) Electrostatic field from voltage and geometry.

(3) System temperature.

(4) Vane null position with no pressure differential.

(5) Approximate pressure on the back of the vane.

The above assumes steady state operation. In nonequilibrium situations, the accuracy of the gauge would suffer because of a net gas flow onto or off of the surface of the vane.

From basic mechanics the torsion of the suspension fiber due to a torque is

$$\theta = \frac{2\ T\ell}{\pi r^4 M}$$

where T is the torque

ℓ is the effective length of the fiber

r is the radius

M is the torsional rigidity.

To relate pressure and force per unit area

10^6 dynes/cm^2 is approximately equal to 760 Torr

or $1.3 \times 10^3 \ \frac{\text{dynes/cm}^2}{\text{Torr}}$

assuming a .0005" tungsten wire with an effective length of 2 cm, a vane with a 4 cm diameter, and a 5 cm moment arm (as shown in Fig. 2) gives

$$\theta = \frac{2\left(\frac{\pi(4)^2}{4} \times 1.3 \times 10^3 \times 5\right)(2)}{\pi(6.25 \times 10^{-4})^4\ (1.5 \times 10^{12})} P$$

$$\theta = 4.5 \times 10^5 \ P, \text{ where P is in Torr.}$$

Experimenters using optical levers in normal environments are able to detect 10^{-5} radians. Simple auto collimating telescopes ($100) are capable of resolving 10^{-5} radians, while better ones ($800) can resolve 10^{-6} radians. More complicated, but not difficult, techniques[1] can resolve 10^{-10} radians.

---

[1] R. V. Jones, J. Sci. Instr. 38, 37 (1961).

For a pressure of 10^{-8} Torr, the angle through which the arm would move if the electric field were off is

$$= 4.5 \times 10^{-3} \text{ radians}$$

$$= .26 \text{ degrees}$$

It is obvious then that the theoretical sensitivity of the proposed device is adequate.

The natural noise limit due to Brownian motion is 10^{-12} Torr and also is not a serious limitation.[2]

The attachment of the tungsten fiber can be accomplished by electroplating copper over the wire.[3]

A .0005 inch tungsten wire will support approximately 10 gms which is sufficient for the vane shown.

On November 10, 1966, a device was tested which had all of the essential features outlined above. The sensitivity was $S = 4 \times 10^{-4}$ or approximately 2 degrees at 10^{-6} Torr. The device was stable, easily damped, and unaffected by the rather bad vibration present in the test system. It was surprisingly rugged and no particular construction problems were encountered.

---

[2] G. Ising, Phil. Mag. <u>1</u>, 827 (1926).

[3] D. W Bassett and A. J. B. Robertson, J. Sci. Instr. <u>36</u>, 321 (1959).

III. A Laboratory Study of Ionization Gauges Related to Spurious Effects Found in Upper Atmospheric Measurements

During a visit to Goddard Space Flight Center on July 26, 1966, George Newton presented several pressure measurement problems of interest to his group. These are relevant to the accurate determination of conditions in the upper atmosphere measured by satellite or rocket probes. It was decided that the vacuum group at the Coordinated Science Laboratory would collaborate on certain experiments to aid in making more accurate low pressure measurements under space conditions.

The final selection of experiments, ranked roughly in order of their importance, was:

(1) Ionization gauge pressure indications in gas mixtures, particularly for the NASA flight gauge types.

(2) Evaluation of surface ionization effects occurring on the grid of the flight B-A gauge. This would include behavior at different grid currents and duty cycles.

(3) Relative sensitivities of various ionization gauges for different gases commonly usable in the laboratory. This question arose because published sensitivity ratios for noble gases agreed well with cross section ratios, but active gases do not.

Basic to both problems (1) and (3) is data on ionization cross sections for gases at the electron energies found in gauges. Therefore, before starting experimental work, a brief literature search on ionization gauge calibration and measurements of absolute ionization cross sections was made, and an attempt was made to evaluate the worth of the various published data. This collected information has considerable value and, in fact, has given tentative answers to some of the problems listed above. A table of data and a bibliography follow. The collection of similar information is continuing, and the final report will be as complete as possible in listing pertinent literature.

TABLE I

IONIZATION GAUGE AND IONIZATION CROSS-SECTION RATIOS (NITROGEN SET EQUAL TO 1.00)

	A	He	Ne	H_2	N_2	Kr	Xe	Hg	CO	O_2	CO_2	Dry Air	H_2O
Conventional Gauges													
1) FP-62	1.12	.157	.221	.437	1.00	1.82	2.45	3.07					
FP-62	1.20	.160	.246	.478	1.00	1.91	2.96	3.30					
VG-1	1.24	.158	.255	.475	1.00	1.81	2.79	3.95					
2) Commercial (unknown)				.532	1.00				1.06	.852	1.36		.894
Metson (suppressor)				.522	1.00				1.03	.769	1.39		1.22
Metson (suppressor)				.523	1.00				1.11				
3) 507	1.07	.190		.382	1.00					1.14		.810	
4) FP-62	1.23	.135	.236		1.00								
3 to 7 gauge average	1.73	.136	.209		1.00								
Inverted Triodes													
5) 5966	1.5	.21	.33	.42	1.0								
—	—	—	—	—	—	—	—	—	—	—	—	—	—
Ionization Cross Section Ratios													
6) 100 V electrons	1.11	.16	.23	.38	1.00			2.12					
7) 120 V electrons	1.24	.123	.282										
8) 120 V electrons				.314	1.00				1.06	1.02			
9) Omegatron sensitivity ratios	1.18	.163		.363	1.00				1.07	1.1	1.27		6.55
10) 120 V electrons	1.10	.134	.269	.309	1.00	1.70	.253			1.07			
11) 120 V electrons	1.11	.147	.287	.348	1.00	1.61	2.16		1.05	1.08	1.41		

REFERENCES FOR TABLE I

1. Dushman and Young, Phys. Rev. 68, 278 (1945).
2. Wagener and Johnson, J. Sci. Instr. 28, 278 (1951).
3. Riddiford, J. Sci. Instr. 28, 375 (1951).
4. Reynolds, Physics 1, 182 (1931) (now J. Appl. Phys.).
5. Schulz, J. Appl. Phys. 28, 1149 (1957).
6. Langmuir and Jones, Phys. Rev. 31, 357 (1928).
7. Smith, Phys. Rev. 36, 1293 (1930).
8. Tate and Smith, Phys. Rev. 39, 270 (1932).
9. Klopfer and Schmidt, Vacuum 10, 363 (1960).
10. Schram, Moustafa, Schatten, DeHeer, Physica 32, 734 (1966).
11. Rapp and Englander-Golden, J. Chem. Phys. 43, 1464 (1965).

See Also:

Kieffer and Dunn, Rev. Mod. Phys. 38, 1 (1966).

Brombacher, "A Survey of Ionization Vacuum Gauges and Their Performance Characteristics," N.B.S. Technical Note 298 (1967).

Brombacher, "Bibliography and Index on Vacuum and Low Pressure Measurement," N.B.S. Monograph 35 (1961).

Kieffer, "Bibliography of Low Energy Electron Collision Cross Section Data," N.B.S. Miscellaneous Publication 289 (1967).

Barnett, et al., "Bibliography of Atomic and Molecular Processes for 1963," Oak Ridge National Laboratory-Atomic and Molecular Processes Information Center (1965).

Barnett, et al., "Bibliography of Atomic and Molecular Processes for 1964," Oak Ridge National Laboratory - Atomic and Molecular Processes Information Center (1966).

IV. Procedure

Since the facilities of the Laboratory do not permit making accurate comparisons of gauges in an absolute sense, it was decided to limit the work to making relative comparisons. Good relative data should be sufficient to answer the questions posed by Newton. The first problem considered before starting the design of the apparatus was the choice of the instrument to be used as the relative standard.

An examination of the data on the table shows that the sensitivities of the omegatron seem to be in good agreement with cross section data. It is also well known that the omegatron has little effect on the system under measurement. These results should not be surprising since the omegatron has a monoenergetic electron beam, and the beam current is small. Some omegatrons are also made of platinum alloy to reduce the effects of changing surface potentials, as well as chemical effects. The properties of the omegatron have been well studied, particularly by Klopfer.[4] Used as a spectrometer, the omegatron would be applicable to the measurement of gas mixtures (Problem 3). However, the omegatron is somewhat unique as a spectrometer in that it can also be used as a total pressure gauge, meanwhile preserving its basic properties of freedom from spurious effects.[5]

Thus, the system for studying these gauge effects could consist of a high conductance manifold carrying the omegatron and the desired

[4] A. Klopfer and W. Schmidt, Vacuum 10, 363 (1960).

[5] A. Klopfer, Transactions of the Am. Vac. Soc. 1961, p. 439, Part I.

ionization gauges. The system should be capable of attaining pressures in the low 10^{-10} Torr range, and should have a gas admission system. Besides the NASA flight gauges, the manifold should have other gauges for comparison, such as the normal Bayard-Alpert Westinghouse type 5966, and Scheumann gauges. The latter is useful because it is dependable at low pressures and is also less subject to surface ionization effects. Figure 3 is a photograph of the system.

In order to select the most suitable type of omegatron for comparison, a small system was set up and several omegatrons tested. One tube from those on hand was selected, and a range of operating parameters determined.

Since it is desired that the comparisons be as accurate as possible, errors in ion current measurement should be held to about 1%. A digital voltmeter will be used to monitor the output voltages from the electrometers. This is more accurate than the panel meter. A Keithley picoampere current source belonging to the laboratory was returned to Keithley for standardization. With its help the required current measuring precision can be maintained.

V. Status - Vacuum Instrumentation

To lessen the chances of damaging the calibrated flight type gauges, the system has been tested without them. Pressures as low as 5×10^{-10} Torr (probably the helium limit) have been reached, and the other gauges must now be attached. The electrometers have been calibrated and, where necessary, input resistors changed to make them consistent. Omegatron control circuits and other control panels have been built and tested. One function of some of the circuits is to prevent damage to calibrated gauges.

VI. Future Work

With all gauges in place and the system at low pressures, the actual measurement program will begin. It should be possible to obtain data for all three problems of Section III simultaneously. The planned sequence of operations is as follows.

With the desired gas bottles on the system, it will be baked and the gauges outgassed. Using a Granville-Phillips pressure controller and a monitor gauge, the pressure will be increased gradually from the base pressure to a limit of about 10^{-6} Torr, with readings made at many points for all gauges under dynamic equilibrium conditions. After doing this for one gas of a pair, it will next be done for the other gas, and then for several mixtures and pressures of the pair of gases. Preparation for different gases will require taking the gas manifold to atmosphere and installing the gas bottles. Gas combinations that are proposed for test are (N_2, Ne); (N_2, A); (N_2, H_2); (A, H_2); and last, more reactive gases. Data analysis will require considerable effort, partly because of the amount of data to be recorded.

VII. Surface Physics

The studies of the adsorption and desorption of gases on metal surfaces have included the study of the adsorption of CO, H_2, N_2, and O_2 on polycrystalline and single-crystalline tungsten surfaces. The single-crystalline planes studied have been the (111) and (110) orientations. These measurements include studies of adsorption from liquid nitrogen temperatures to high temperatures. Substantial information concerning the spectrum of adsorption energies for these gases has been obtained. We have found that, in general, one is not able to apply simple kinetics to the results obtained. This is a result of the fact that particles are not bound with a discrete energy but that the effective binding energy is a function of coverage. Calculations of desorption rates for such systems can be correlated with the experimentally measured desorption rates.

This work is in preparation for a thesis and for publication. When this is completed, these documents will be submitted as a part of the final report for this grant.

Publications:

Dallos and Steinrisser, J. Vac. Sci. Technol. 4, 6 (1967).

Steinrisser, J. Vac. Sci. Technol. 4, 44 (1967).

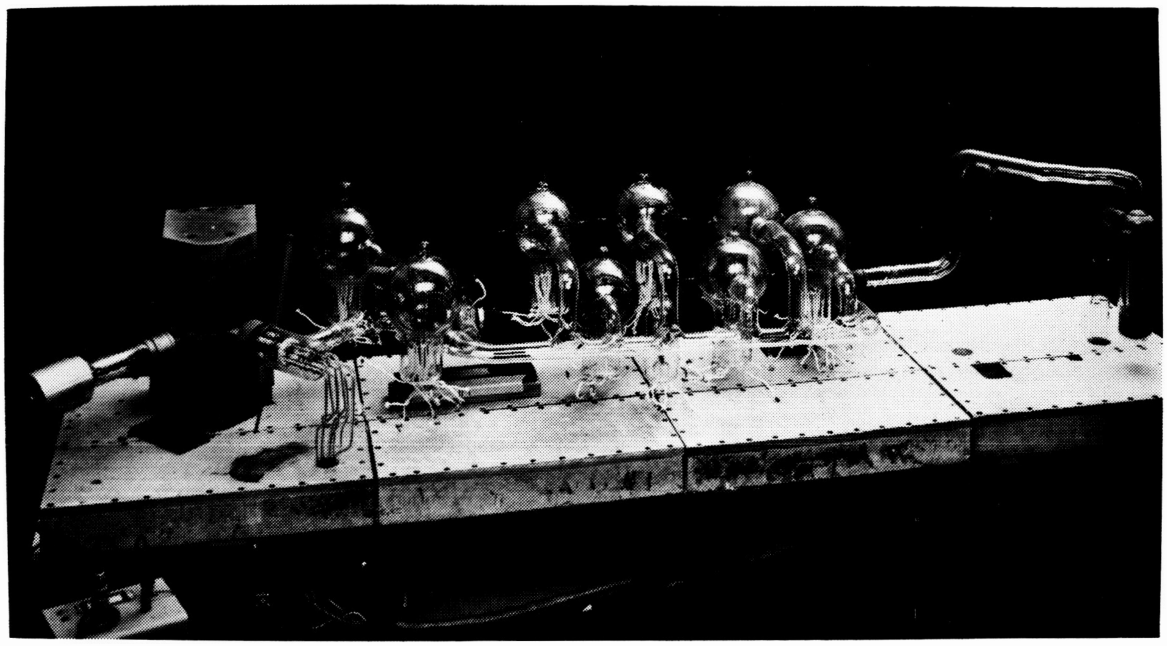

Fig. 1 Schuemann, Bayard-Alpert, and magnetron type gauges with mass spectrometer on UHV system.

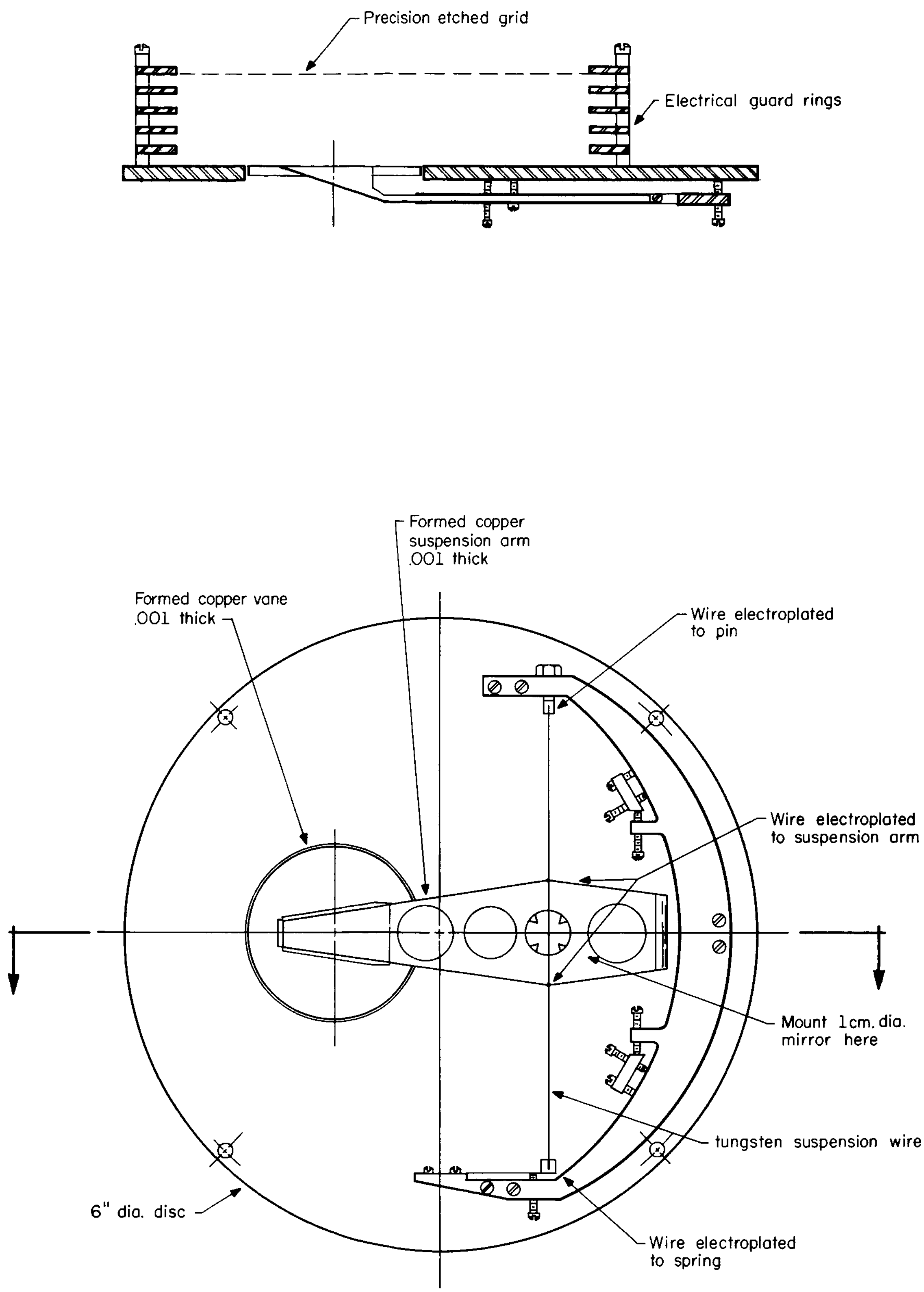

Fig. 2 Low Pressure Absolute Manometer.

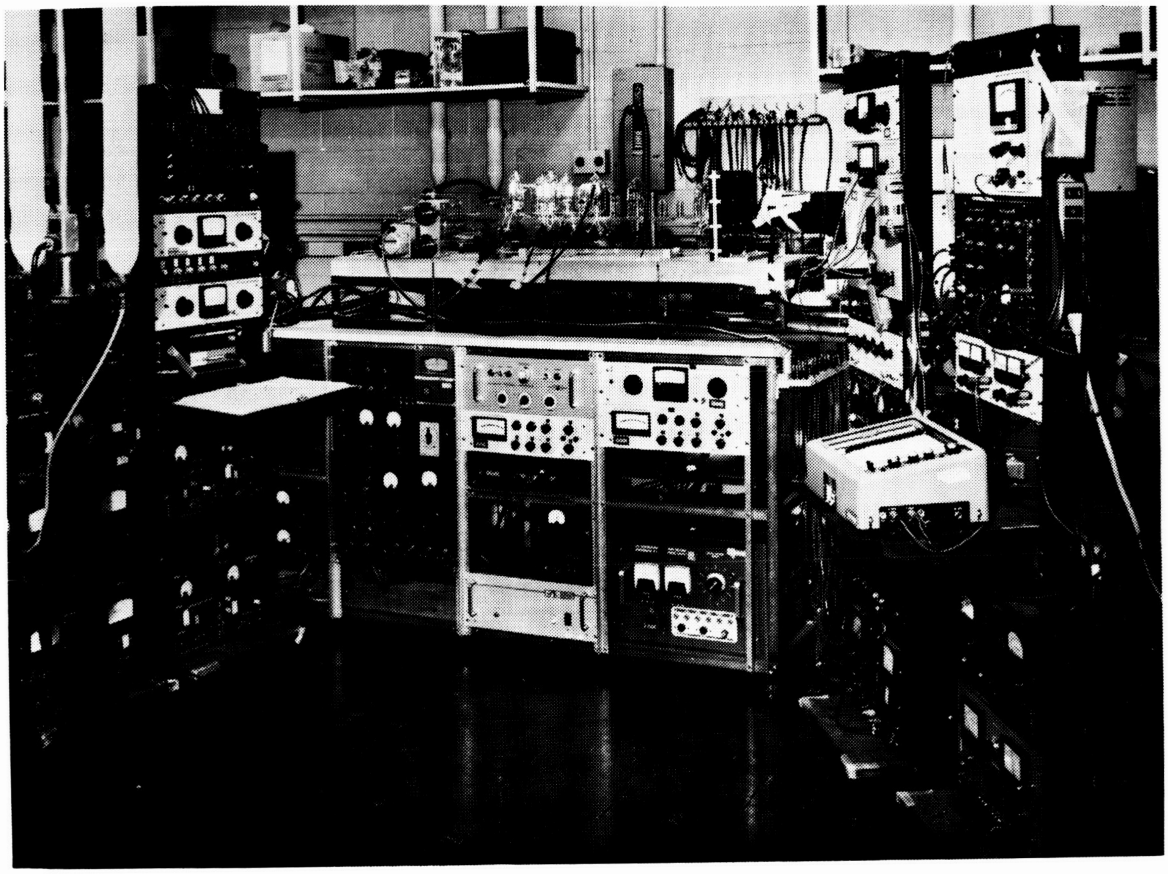

Fig. 3 Gauge Comparison System and Associated Equipment.